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Propellant/Material Compatibility Program and Results

Ten-Year Milestone

Clifford Moran Roy Bjorklund

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July 15, 1982

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National Aeronautics and Space Administration

Jet Propulsion Laboratory California Institute of Technology Pasadena, California



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Jet Propulsion Laboratory California Institute of Technology Pasadena, California The research described in this publication was carried out by the Jet Propulsion Laboratory, California Institute of Technology, under contract with the National Aeronautics and Space Administration.

CONTENTS

I.	Objectives	1.
II.	Pretest Activities	2
111.	Posttest Analysis	4
	A. Posttest Chemical Analysis	4
	3. Posttest Specimen Analysis	4
IV.	Material Ratings	10
	Λ. Hydrazine/Materials, 10-Year Compatibility Ratings	1. 0
	B. Monomethylhydrazine/Materials, 10-Year Compatibility Ratings	1.3
	C. Nitrogen Tetroxide/Materials, 10-Year Compatibility Ratings	14
v.	Conclusions	16
	A. Nydrazine	16
	B. Monomethylhydrazine	16
	C. Nitrogen Tetroxide Mon-1	16
Gloss.	ary of Terms	17
Refer	ences	18
Appen	dix A. Summary of Specimens Remaining in Storage	19
Appen	dix B. Pretest Propellant Assays	25
Appen	dix C. Summary of Posttest Analyses and Results	29
Appen	dix D. Scanning Electron Microscope Examination of Specimens	47

1 1801 03		
1.	Procedure for capsule filling	3
2.	Procedure for posttest chemical analysis, hydrazine	5
3.	Procedure for posttest chemical analysis, monomethylbydrazine	6
4	Procedure for posttest chemical analysis, nitrogen tetroxide	7
5.	Procedure for metallic specimen analysis	8
6.	Materials rating system	11
D-1.	Specimen 0018, 6061-T6A1, 500X. Unbroken surface film	48
D-2.	Specimen 0016, 6061-T6A1, 500X. Cracked and flaking film	48
D-3.	Specimen 1130, 304L, heat-affected zone, 500X. Thin irregular film	49
D-4.	Specimen 0896, Ti6A1-4V, 500X. Thin irregular film, minor cracking and pitting	49
D-5.	Specimen 1499, 6061-T6A1, 500X. Thin film with cracking and flaking	50
D-6.	Specimen-1527, 304L, 500X. Unbroken surface film	50
D-7.	Specimen 0073, 2014-T6A1, 500X. Thin-to-moderate film showing evidence of erosion, not surface pitting	5
D-8.	Specimen 3155, 304L, 500X. Surface virtually untouched	51
D-9.	Specimen 1975, 347, 500X. Corrosion at area of highest stress .	52
D-10.	Specimen 0191, Ti6A1-4V, 500X. Unbroken surface film	52
Tables		
A-1.	Specimens in hydrazine (MIL-P-26536B)	20
A-2.	Specimens in purified hydrazine (VL-75)	21
A-3.	Specimens in hydrazine-hydrazine nitrate	22
A-4.	Specimens in monomethylhydrazine (MIL-P-27404A)	23
A-5.	Specimens in nitrogen tetroxide (MON-1) (MSC-PPD-2B)	24
B-1.	Assay of hydrazine	26

Tables		
B-2.	Assay of monomethylhydrazine used in material compatibility 'est program	27
В-3.	Assay of nit.ogen tetroxide used in material compatibility test program	28
C-1.	Summary of posttest analyses and results - hydrazine	30
C-2.	Detailed posttest analyses and results - hydrazine	36
C-3.	Posttest analyses of hydrazine controls	37
C-4.	Summary of posttest analyses and results	38
C-5.	Detailed posttest analyses and results - monomethylhydrazine	40
C-6.	Summary of posttest analyses and results - nitrogen tetroxide (MON-1)	41.
C-7.	Detailed posttest analyses and results - nitroger cetroxide	44

ABSTRACT

This report details the analyses and results of a test program to establish the effects of long-term (10 years or more) contact of materials with earthstorable propellants for the purpose of designing chemical propulsion system components which can be used for current as well as future planetary spacecraft. This report covers the period from the publication of JPL TM 33-779 in 1976 through the testing accomplished in 1981.

The following propellants are reported herein: hydrazine, monomethyl-hydrazine and nitrogen tetroxide. Materials included the following: aluminum alloys, corrosion resistant steels and a titanium alloy. The results of the testing of more than 80 specimens are included. Material ratings relative to the ten-year milepost have been assigned.

Some evidence of propellant decomposition was found. Titanium is rated as acceptable for ten-year applications. Aluminum and stainless steel alloys are also rated as acceptable with few restrictions.

I. OBJECTIVES

The basic objective of the NASA propellant/material compatibility program is to obtain detailed compatibility data for periods up to 10 years or longer. Concommitant objectives are the development of (1) a standard test methodology, (2) basic procedures for compatibility testing and analysis, and (3) a system of rating the materials for compatibility in long-term applications.

The scope of the test program was established to serve the needs of long-term planetary spacocraft. The "real-time exposure testing" approach was chosen as the most productive method of determining the acceptability of materials of construction for chemical propulsion systems.

The test program is comprehensive. Materials are tested unstressed and stressed in each propellant. Groups of materials are tested in (1) bimetal separated, (2) bimetal contact, (3) welded, (4) brazed, (5) plated & id (6) coated configurations. In addition there are control units containing only propellants to monitor the behavior of the propellant itself.

A summary of the specimens remaining in storage is presented in Appendix A. The metals include aluminum alloys, corrosion-resistant steels and titanium alloy. Nonmetals include fluorocarbon coating or grease, ethylene propylene terpolymer and butyl rubber compound elastomer. Whenever possible, these were procured and certified to meet military specifications or equivalent specifications used in aerospace applications. A detailed description of these is given in Ref. 1. The emphasis has been on materials appropriate to small liquid-propellant thrusters. Most of the materials originally placed into storage were those typically used in the Viking Orbiter 1975 propulsion system.

11. PRETEST ACTIVITIES

Preparation of specimens and the assembly of the test units are detailed in Ref. 1. The procedures used for capsule loading are outlined in Fig. 1. The pretest propellant assays are given in Appendix B.

The test container was made of borosilicate glass ("Pyrex"). The design was such that it could be completely scaled with the propellant and specimen in contact with only the glass. Internal pressure in the glass capsules is monitored by attached strain gauges. Earlier capsule designs utilizing an attached bourdon gauge had the disadvantage of propellant interaction with gauge material.

Capsule loading was accomplished under clean room conditions. The specimen was inserted into the capsule and a glass transition section fused to the capsule opening. Propellant was admitted either by the use of a glass syringe or by distillation. The propellant was frozen; the nitrogen used to blanket the propellant was evacuated, and the capsule tipped off by fusing the glass.

After completing the final seal, the capsules with specimens in place were maintained with the propellant frozen. This allowed for safer handling and shipping, and also inhibited any premature chemical reaction. The capsules were shipped to ETS and placed in storage in a special test facility held at a constant temperature of $43^{\circ} \pm 1^{\circ} \text{C}$ (110 $\pm 2^{\circ} \text{F}$).

Under the general program plan, a few test units were due to be removed semiannually for posttest analysis. However, this schedule was not adhered to, and capsules were removed on an irregular basis. The criteria for removal from test were (1) to obtain periodic data points for certain, widely used metals, e.g., CRES 304L, (2) to analyze those test units in which the propellant was discolored, thereby indicating a high degree of corrosion, and (3) to remove from test for safety reasons those units indicating pressures greater than 34.4N/cm²(50 psia). The last criterion proved unsatisfactory because the strain gauges often gave spurious results, especially as they aged.

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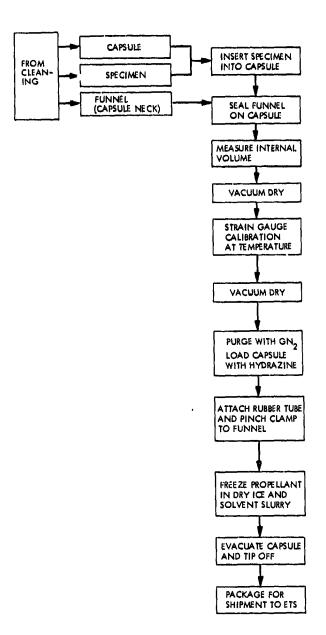


Fig. 1. Procedure for capsule filling (strain gauge type)

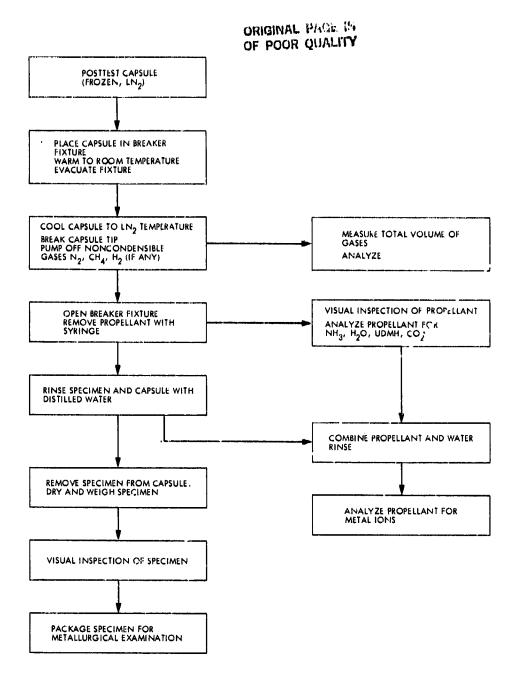


Fig. 3. Procedure for posttest chemical analysis, monomethylhydrazine

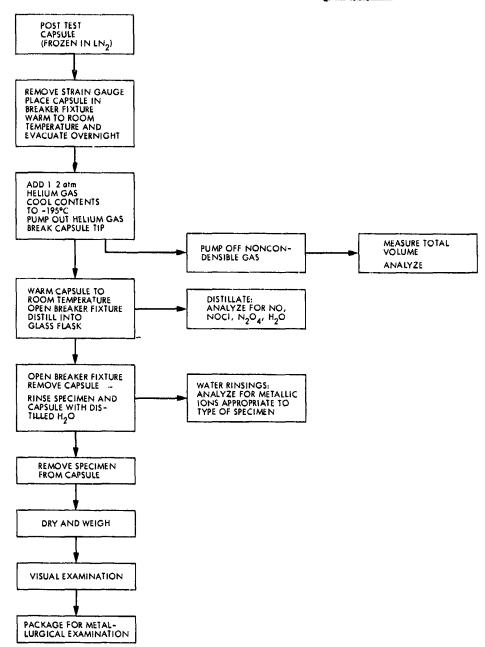


Fig. 4. Procedure for posttest chemical analysis, nitrogen tetroxide

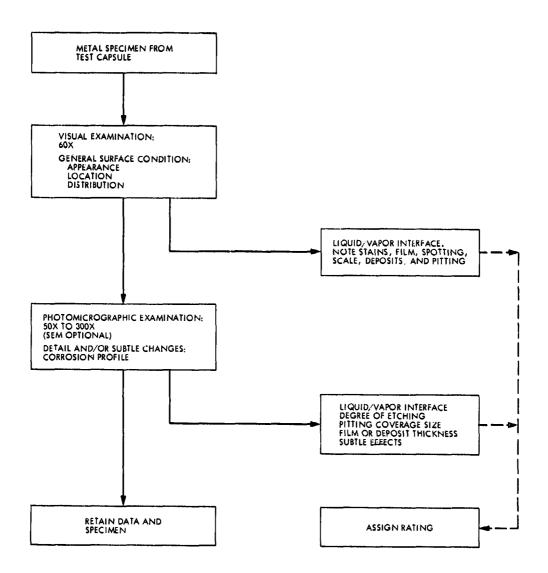


Fig. 5. Procedure for metallic specimen analysis

The important observations are listed:

- (1) Weight.
- (2) Appearance and location of the liquid/vapor inverface boundary.
- (3) Presence and distribution of deposits, stain or film.
- (4) Streaks, mottling or spotting of surfaces.
- (5) Crystalline deposits.
- (6) Flaking and/or cracking of films.
- (7) Extent and location of etching.
- (8) Extent and location of pitting, size of pits.
- (9) Extent and location of cracking.

IV. MATERIAL RATINGS

The criteria for the determination of long-term propellant/material compatibility ratings are fully covered in Ref. 1. The general scheme for determining the final ratings is shown in Fig. 6. The materials rating system has been established to derive engineering information and design guidelines for JPL test programs. The system provides for:

- (1) A basis for selecting structural or component material candidates based on the application and operating environment.
- (2) A basis for rating the acceptable materials in terms of the best data currently available.

The classification and symbols used are:

- A = acceptable
- N = not acceptable
- I = incomplete compatibility data with respect to conditions or duration
 of exposure
- R = restricted compatibility due to corrosion of materials or propellant degradation at conditions that could influence the mission, component or operating specifications.

A. HYDRAZINE/MATERIALS, 10-YEAR COMPATIBILITY RATINGS

Based upon the rating system discussed in the previous section, the compatibility ratings for materials in contact with hydrazine—for 10 years at 43°C (110°F) are summarized as follows:

Material	Rating	Qualification
Propellant only	A	R
Aluminum alloys 6061-T6, 2014-T6	A	R
Corrosion resistant steel 302 and 303	N	
304L, 316, 347, 17-7 430, 446, 355	A	R
350	N	I
Titanium alloy 6Al-4V	Α	

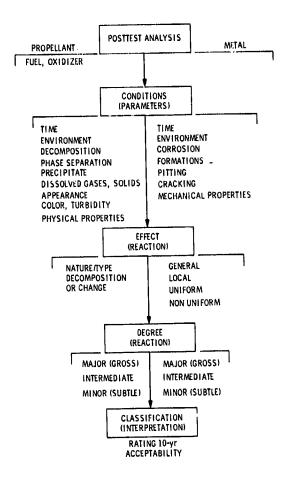


Fig. 6. Materials rating system

Material	Rating	Qualification
Others		
Braze	Λ	1
Gold plate	Α	i
Polymers		
AFE-332	Α	Ţ

1. Hydrazine

Decomposition of the propellant resulted mainly from contaminants in solution and the catalytic effects of metals, and, generally, the rate of decomposition was acceptably low, i.e., less than 0.1% per year for both the MIL-P-26536B specification grade and the purified hydrazine. The hydrazine rating is restricted only in presence of contamination and/or catalyzing metal ions.

2. Aluminum Alloy, Type 6061-T6, 2014-T6

During analysis of this aluminum alloy, the main features noted were moderate corrosion and the formation of a surface film, some of which flaked off. The film did not appear to catalyze propellant decomposition. The aluminum alloy rating is restricted because of the possibility of oxide layer flaking and plugging filters.

Corrosion-Resistant Steel, Types 302 and 303

All capsules with these alloys were found to contain excessive pressure... These CRES alloys are rated not acceptable because of excessive catalysis of the propellant.

Corrosion-Resistant Steel, Types 304L, 316, 347, 17-7, 430, 476, 355, 301

These specimens were found to contain some random corrosion and discrete pitting, with only a thin film formation. A quantity of iron was transferred to the solution, and this can catalyze hydrazine decomposition. However, except for a few anomalies, e.g., the 347 weld stress and a 304L weld-stress, the hydrazine decomposition rates were low. These CRES alloys are restricted only because of possible contamination problems and possibly a catalytic interaction in weld-stress specimens.

5. Corrosion-Resistant Steel, Type AM 350

This alloy produced unacceptably high levels of propellant decomposition and is rated not acceptable. However, the rating is considered incomplete because of insufficient data.

6. Titanium Alloy, Type 6A1-4V

The general effects noted in this alloy were the Irregular formation of corrosion products on the surfaces — particularly in the vapor exposed area — and minor pitting. Film buildup was very thin. Apparently none of the titanium dissolved in hydrazine, and the catalytic activity was very low.

7. Others

- a. Braze. A 301 Cryo/3041 tank-to-diaphragm weld showed no evidence of corrosion; however, the rate of propellant decomposition was 0.2% per year. This alloy is given an acceptable rating; however, these are incomplete data because only one specimen was tested.
- b. Gold plating. Imperfections (pitting or porosity) in the plating allowed the propellant to interact with the substrate. This produced nonuniform blisters and to a minor extent, affected the bonding integrity of the plating to the metal. The acceptable rating is incomplete because of insufficient data.

8. Polymeric, Type AFE 332

No serious degradation of this polymer was observed after a three-year exposure, even in the stressed configuration. The acceptable rating is incomplete because data is lacking for 10-year period.

B. MONOMETHYLHYDRAZINE/MATERIALS 10-YEAR COMPATIBILITY RATINGS

Compatibility ratings for materials in contact with monomethylhydrazine for 10 years at 43°C (110°F) are summarized as follows:

Material	Rating	Qualifier
Propellant only	۸	
Aluminum alloy		
6061-T6	Α	1
Corrosion-resistant steel		
304L, 347	Λ	
Titanium alloy		
6A1-4V	Α	Ī

1. Monomethylhydrazine

The rate of decomposition of this propellant in contact with any of the materials tested was acceptably low for the 10-year period, i.e., less than 0.1% per year.

2. Aluminum Alloy, 6061-T6

The primary effect noted on the surface of this alloy was a very thin, even oxide coating which did not appear to flake. Although up to 100 ppm of aluminum was found to be dissolved in the propellant, no catalytic effect was noted. The acceptable rating is incomplete because at this point only two specimens have been tested.

3. Corrosion-Resistant Steel, Types 304L and 347

Both types of stainless steel alloys exhibited only minor corrosion with a surface film formation. Considerable iron was dissolved (100 ppm) but the catalytic effect was small.

4. Titanium Alloy, Type 6A1-4V

Corrosion of this alloy was light, but corrosion products were deposited in the vapor-exposed area and at the liquid-vapor interface. A small quantity of very fine sediment was found in a capsule that also contained chloride contamination. The acceptable rating is incomplete because only two specimens have so far been tested.

C. NITROGEN TETROXIDE/MATERIALS 10-YEAR COMPATIBILITY RATINGS

Compatibility ratings, for materials in contact with nitrogen tetroxide MON-1 for 10 years at 43°C (110°F) are summarized as follows:

Material	Rating	Qualifier
Propellant only	A	
Aluminum alloy 6061-T6	A	R
Corrosion-resistant steel 302, 303, 304L, 316, 32L, 347, 17-4, 17-7	A	R
Titanium alloy 6A1-4V	A	
Nickel	N	

1. Nitrogen textroxide MON-1

This propellant was found to be stable when in contact with any of the materials tested.

2. Aluminum Alloy - 6061-T6

The primary effects noted in this alloy were corrosion and formation of a surface film. Corrosion was minor because of the protective nature of the film. The rating of this alloy is restricted only because of the possibility of the oxide layer flaking and plugging filters.

Corrosion-Resistant Steel, Types 302, 303, 304L, 316, 321, 347, 17-4

In all CRES alloys, only very minor corrosion was seen. However the rating is restricted due to the iron nitrate dissolved from the surface of the metal with possible flow decay problems in certain applications.

4. Titanium Alloy, Type 6A1-4V

Only minor corrosion was seen in this alloy, usually as a spotty grey appearance. Oxide formations were nonuniform and showed signs of cracking, but not flaking. Thin film appeared to adhere tenaciously and did not dissolve in the propellant.

5. Other: Nickel

Extensive corrosion was observed with flaking of the oxide coating leaving a heavily etched surface. The propellant also contained dissolved nickel.

V. CONCLUSIONS

A. HYDRAZINE

Under the controlled environmental conditions, and in the absence of metal surfaces and degrading contaminants, the rate of decomposition of hydrazine in these tests was determined to be 0.015% per year. In the presence of metal specimens, the decomposition of hydrazine was within an order of magnitude of the rate of the propellant control samples, i.e., less than 0.15% per year. The few exceptions to this were all CRES alloys and the reasons for the greater rate of decomposition were not readily apparent. In general, however, none of the tested materials would, with few exceptions, excessively catalyze the decomposition of hydrazine during long-term (10-year) missions.

B. HONOMETHYLHYDRAZINE

The rate of decomposition of propellant, MMH, in those tests was quitesmall: 0.02 to 0.10% per year. The corrosion products of aluminum appeared to cause only an insignificant amount of decomposition, whereas the metal ions or corrosion products of CRES alloys or titanium seemed to catalyze the decomposition somewhat more.

The dissolved iron from CRES was higher in MMH than is generally seen in the hydrazine propellants, but the catalytic effect of this ferric ion was obviously less with MMH. Even the highest concentration of iron — 100 ppm — yielded a decomposition of only 1.1% in 11 years. It would appear that the materials tested would not excessively catalyze the decomposition of monomethylhydrazine during long term (10-year) missions.

C. NITROGEN TETROXIDE MON-1

No evidence of propellant decomposition has been found. Given the chemical nature of NTO, about the only change possible would be disproportionation into various other oxides of nitrogen, and this disproportionation usually requires more severe conditions (very much higher temperature) than were present in these tests. The CRES alloys, however, did provide enough dissolved iron to cause a possible flow decay problem in certain systems.

CLOSSARY OF TERMS

AES auger electron spectroscopy

CRES corrosion-resistant steel

ELA extra-low interstitial grade

EPT ethylene-propylene terpolymer

ETS JPL Edwards Test Station

FEP Fluorinated ethylene-propylene polymer

MMI monomethylhydrazine

MON-1 mixed oxides of nitrogen (NTO with 1% NO)

NTO nitrogen tetroxide

SEM scanning electron microscope

SIMS secondary ionization, mass spectroscopy

TFE tetrafluoroethylene

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Table A-1. Specimens in hydrazine (MIL-P-26536B)

Material	Stug	Bimetal contact	Bimetal separated	Stressed (stressed to 67% of yield)	Welded	Coatings	Controls	Total
		W/30HL W/347 W/2219-T81 W/6061	W/347 W/6061 W/6-4Ti	Slag Weld Birnetal weld	Self	Gold plate Chrome plate Anedize Krytov 240 AC		
304L	3				1			4
316	1	1	1 1	1	!			4
347			1 1		[2
430	1			ļ	!	3		4
446						2		2
17-4			ł			1		i.
17-7	1		1	Ì		1	Ì	2
355	1	<u> </u>						1
356-T6A1						2		2
2014-T6			1	5		1 2		8
2219-T81	2				1			2
5052						2 2		4
6061T6	13	2 1 5	ı	3 1		2 3 3		34
70 75 T6						1		 _
6AI-4V Ti	27	2	2	14 16		8	Į	69
6AI-4V ELI	3		<u> </u>	6	ļ	<u> </u>	 -	5
Nłobium			1			2		2
EPT-10	2	2 3 4	1		ŀ		i	11
Tefion LRV-448	2				1	<u> </u>	1	2
N ₂ H ₄							28	28
Total	56	2 4 3 2 9	4 1 2	28 17	1	2 5 6 22	28	192

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Table A-2. Specimens in purified hydrazine (VL-75)

Material	Slug	Bimetal contact	ntact of yield)		(stressed to 67% of yield)		(stressed to 67% of yield)		(stressed to 67% of yield)		(stressed to 67% of yield)		(stressed to 67% of yield)		(stressed to 67% of yield)		(stressed to 67% of yield)		Wel	deđ	Brazed	Controls	Total
		W/64Ti	Slug	Weld	Bimetal weld/17-4 PH	Bimetal weld/347	Self	Bimetal/304L	Self														
301 CRYO								2			2												
3041. CRES	1		3	3	2	1	3				13												
316									1		1												
446 -	\			4			1				5												
6Al-4V Ti	1		ì								2												
AFE-332	4	26									30												
EPT-10		6									6												
N ₂ H ₄										4	4												
Total	6	32	4	7	2	1	4	2	1	4	63												

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Table A-3. Specimens in hydrazine-hydrazine nitrate

Material	Slug	Bime conta 1909/X		Stress (atress to 67 of yie	ed %	Welded Fig.	Total
6061-T6 AI		.,	1	1		2	4 -
6Al-4V Ti	1	1		3	3	4	12
Total	1	1	1	4	3	6	16

Material	Slug	Bimetal contact		(strei to 6	Stressed (stressed to 67% of yield)		(stressed to 67%		(stressed to 67%		Brazed	Controls	Total
		W/347	1909/M	W/6-4Ti	Slag	Weld	Self	Self					
303 CRES	3	2	2	1	2	2		2		14			
304L	2	2	2	2	}	2	1	}		11			
316	3	2-	4	2	2	2	2	ļ	}	17			
347	2		2	2	ı	2	2			11			
6061-T6 Al	3	1			1	2	1	į		8			
6Al-4V Ti		2	2		3	3	3)		13			
Teflon-FEP	2	ł			l		ł		1	2			
Teflon-TFE	4							<u> </u>		4			
MMH (control)								·	2	2			
Total	19	9	12	7	9	13	9.	2	2	82			

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Table A-5. Specimens in nitrogen tetroxide (MON-1) (MSC-PPD-2B)

Materia!	Slug		Bimetal contact		Stree (stree to 6 of yi	ssed 7%	Welded		Coatings		Controls	Total
		W/347	W/6061	W/6-4Ti	Sing	Weld	Self	Chrome plate	Anodize	Krytox 240 AC		
302 CRES	3	1	1		1			_				6
303	4	2										6
304L	4	2			1		1			2		10
316	3											3
321	3	1		,	1		2					7
347	2		7	3	2		,	2		2		18
416	3	1										3
17-4	4									1		5
17-7	3	<u> </u>								2		5
5052Al	4				ĺ							4
606lT6	2	2		4	4				4	3		19
7075	3							ļ				3 -
6Al4VTi	8	}			16	8				3	•	35
Nickel	5									2		7
N ₂ O ₄	<u> </u>										5	5
Total	51	8	8	7	25	8-	3	2	4	15	5	136

Appendix B

Pretest Propellant Assays

ORIGINAL PAGE 13 OF POOR QUALITY

Table B-1. Assay of hydrazine

Constituent of property	MIL-P-26536B ^a specification limits	Drum H-3155 ^b	Purified hydrazine ^C
Hydrazine assay, % by weight	97.5 min	98.7	99.2
Water, % by weight	2.5 max	0,50	0,60
Density at 298 K (77°F), g/cm ³	1.004 ±0.002	1,004	1.004
Particulate, µg/cm ³	10 max	4,6	0,5
Ammonia, % by weight	Not required	0.07	0,06
Aniline, % by weight	Not required	0,25	<5 ppm ND ^d
Carbon dioxide, µg/g (ppm)	Not required	35	19
Nonvolatile residue (ash) µg/g (ppm)	Not required	60	<5 ND
Dissolved metals, µg/g (ppm)	Not required		
Iron		0.5	0.38
Aluminum		0.05	< 0.05 ND
Nickel		1.10	0.03
Munganese		0.35	0.04
Cobalt		< 0.5 ND	<0.02 ND
Chromium		0.16	0.02
Copper		0.09	0.04
Zine		0.13	0.01
Silicon		<0.5 ND	< 0.5 ND
Magnésium		<0.01 ND	0.01
Sodium		0.20	0,06
Potassium		0.05	0,04
Calcium		0.04	< 0.04 ND
Barium		<0.2 ND	< 0.2 ND
Boron		<10 ND	<10 ND
Dissolved anions, µg/g (ppm)	Not required		
Fluoride		<0.5 ND	< 0.5 ND
Chloride		0.3	< 0.3 ND
Sulfate		<0.5 ND	< 0.5 ND
Nitrate		< 0.5 ND	< 0.5 ND

^aVersion of hydrazine specification in force at the start of program.

^bJPL assay replaces SRI assay (Ref. 2) erroneously reported in Ref. 1.

^cMartin Marietta Corp. Lot No. 51, JPL assay.

dNct detectable by method of analysis used.

Table 8-2. Assay of monomethylhydrazine used in material compatibility test program

Constituent or property	MIL-P-2740A Amendment 2 specification limits	Assay of N2H3CH3 prior to filling capsules
Monomethylhydrazine (N2H3CH3) assay, % by weight	98.3 min	99.42
Water, % by weight	1.5 max	0.48
Particulate, milligram per liter	10.0 max	0.97
Density, grams per milliliter at 77°F (25°C)	0.870 to 0.874	0.872
Ammonia (NH ₃), % by weight	Not required	0.10

Table B-3. Assay of nitrogen tetroxide used in material compatibility test program

Constituent or property	MSC-PPD-2B specification limits	Assay of N ₂ O ₄ prior to filling capsules
Nitrogen tetroxide (N ₂ O ₄) assay, % by weight	98,50	99.36
Nitric oxide (NO) assay, % by weight	0,8 ± 0,20	0,60
Water equivalent, % by weight	0,10 max	0.06
Chloride (NOCI), assay, % by weight	0,08 max	< 0,01 ND
Ash, % by weight	****	< 0.01 ND

Appendix C

Summary of Posttest Analyses and Results

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				Specimen				Propellant	
Specimen	Duration of test, days	Material	Configura-	Remarks	Weight change. mg	Capsule pressure, N/cm ² at 430C	Decomposition.	Remarks	Halide content, ² mg
1000	3939	6061-T6 Al Slug	Sing	Light corrosion; overall thick white film with cracking at the I,V area; light etching and no pitting.	8 .	7.45	0.53	Slight decomposition: water-white.	20
0053	3939	Ti6AJ-4V	Slug	No corrosion; overall thin film; no pitting.	5.1	13.75	88.2	Shight decomposition: water-white.	2.20
0073	4233	2014-T6AI	Slug	Light corrosion; overall thick film; light etching; no pitting	6.9	16.83	1.16	Slight decomposition: water-while.	09.0
1610	3986	Ti6Al-4V ELI	Slug	Light corrosion: overall thick film with cracking; light eaching in the L/V and L area; light pitting in the L/V and V areas approximately 1 x 10 ⁻⁶ in, in diameter with none in the L area.	3.2	3.98	0.35	Slight decomposition: water-white.	0.20
0533A	4038	6061-T6AJ	6061-T6Al Bimetal STD	Light corrosion; thin film in the L and V areas and thick in the L/V area; light etching and no pitting.	1.8	ı	1	Severe decomposition: pale pink.	1.80
06338		302 CRES	Bimetal	Moderate corrosion and thick film in the L/V and L areas: light corrosion and a thin film in the V area; light etching; moderate pitting in the L/V and L areas and none in the V area.	4.				
0559A	4027	Ti 6A14V	Bimetal STD	Light corrosion; overall thick film, with cracking in the L/V area; light etching and no pitting.	1.0	12.99	0.84	Slight decomposition: water-white.	260
0559B		6061-T6Al Bimetal	Bimetal	No corrosion; overall thick film, with cracking in the L/V area; no pitting.	.21				

Table C-1. Summary of posttest analyses and results - hydrazine

ORIGINAL FACT FO

				Specimen				Propellant	
Specimen number	Duration of test, days	Material	Configuration	Remarks	Weight change, mg	Capsule pressure, N/cm ² at 43°C	Decom-	Remarks	Halide content,
0585B	4027	Ti6Al-4V 303 CRES	Bimetal STD	Light corrosion in the L/V and L areas and moderate in the V area: thick film in the L/V and V areas and thin in the L area: light etching: moderate pitting in the L and V areas and none in the L/V. Light corrosion in the L/V and L areas and moderate in the V area; thick film in the L/V and V areas and thin in the L/V and V areas and thin in the L/V and V areas and thin in the L/V and V areas ing: light pitting in the L/V:	0.9	47.55	2.50	Moderate decomposition: pale blue.	30.0
0765	3954	Ti6Al-4V	Slug-stress	Moderate in the V and 1: one in the L area. Light corrosion: overall thick film with eracking in the L/V area: light etching; moderate pitting in the L/V and V areas (1 x 10 ³ in.).	0.2	7.30	0.54	Slight decomposition; water white.	0°0
0767	3954	Ti6A1-4V	Slug-stress	Light corrosion; thick overall film with cracking in the L. V area; light etching; moderate pitting in the L/V and V area; (1 x 10 ⁻⁶ in.) and none in the L area.	-0. <i>S</i>	11.20	0.73	Slight decomposition: water-white,	6.80
1601	4053	6061-T6A1	Slug-anodized	L ight corrosion: thick film overall with cracking in the L/V and V areas: no pitting	-10.2	I. I	0.80	Slight decomposition: white sediment	Š.
1099	2980	6061-T6AI	Slug-gold plated	Severe blistering of surface.	13	11.80	0.73	Slight decomposition, light brown color.	6.9
1853	3689	347 CRES	Slug	No corrosion; thin overall film; no pitting.	0.7	4.76	0.33	Slight decomposition, water-white.	N O

Table C-1 (contd)

ORIGINAL PAGE 13 OF POOR QUALITY

				Specimen	,	,		Propellant	
Specimen	Duration of test, days	Material	Configura- tion	Remarks	Weight change, mg	Capsule pressure, N/cm ² at 43°C	Decom- position,	Remarks	Halide content,
3223 c	2645	304L CRES	304L CRES Sugstress	Light corrosion: thin film on the L/V and V areas and very thin on the V area; light etching and no pitting.	-12.9	245	0.25	Very low decomposition: water-white.	ě
3239	3061	Ti6A1-4V	Slag	Light corrosion; thick overall film; light etching and no pitting.	0.3	2.58	0.19	Very low decomposition: water-white.	Q.
3257	2550	Ti6A1-4V	Slug-stress	No corrosion; thin overall film: no pitting.	-0.2	3.67	0.21	Very fow decomposition:	
3261	1112	AFE-332	"O" rings stressed on a Ti6Al-4V fixture		:	3.86	0.26	Very low decomposition: no residue; water-white,	ŀ
3267	2672	304L CRES	Tube welded	Light corrosion; thick overall film; light etching and no pitting	-1.0	į	;	÷	:
3277	2709	304L CRES/ Weld, stress 304L CRES	Weld, stress	Light corrosion; thin film in the L/V and L areas and thick in the V area; light etching in the L/V and V areas and none in the L area; no piting seen in the weld zone. Light corrosion, thin film in the L/V and L areas and thick in the V area; light etching and no pitting seen in the heat affected zone.	-6.7	47.39	3.73	Moderate decomposition: water white.	9.
3305	2597	301 CRYO/ 304L CRES	Tank/dia- phragm weld	Light corrosion; thin film in the L-Weld and L/V areas and a thick film in the V and V-Weld areas with cracking in the V area; light etching and no pitting.	-5.1	16.31	1.28	Slight decomposition: water-white.	Š
3323	1129	AFE-332	ASTM dog- bone forthe fold in glass sleeve	:	0.3	248	0.22	Very low decomposition	;

Table C-1 (contd)

*Chloride (halide) introduced as contamination during improper cleaning and preparation procedures.

^bPretest specimen weight in error.

Specimens numbered greater than 3200 were exposed to VL-75 grade purified hydrazine.

 $ND \approx none detected;$ less than quantity detectable by analytical technique used.

... = not measured; data not available

L = liquid exposed region.

V = vapor exposed region.

L/V = liquid-vapor interface.

ORIGINAL PAGE 13 OF POOR QUALITY

Harden Change Part Mark Change Chang		Specim	Specimen acigni					Analysis of propenant	and and						-	The same of the sa		
1,500, 0,001, 1,0	Specimen number		Change.	Fc + C. + X1 + Mn. mg		11+ V.	Total Mith		(6) uş g	H20.	Ť	Andens	Funty.	2+H2 00 STP	. VH3.	Tr. St.	# F	7 F
1,250, 2, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0,	7000	1,35.96	0.0018		} :	:	:	7.00		0.24	4.0	0.34	:	37,78	18.13	32.15	กรุก	\$2.75
1798 00003	00S3	3.2636	0.0051	:	;	0.21	0.21	2.20	;	0.39	0.52	0.27		54.45	137.03	37.45	7,32	EX.0.
13.36 0.0031 0.11 0.12	0073	1.8928		9.10	:	:	0.10	0.60	:	0.44	6,75	0,25		47.27	100	£.	Ţ.	14.4
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2.9.12.2 GOODIT OAS (6.9) 6.9	1601	1,9480		:	8,69		8.60	92	:	0,49	5.7			12.59	142,31	22.34	647	16.4
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Table C-2. Detailed posttest analyses and results - hydrazine

Tapsake broke in fixture while warming.

By distance samples tool.

Trotal metals include those listed in parentheses.

ND = more desected.

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Table C-3. Posttest analyses of hydrazine controls

				Analysis of	propellan	t		Analysis	of gas
Specimen number	Duration of test, days	Pressure, N/cm ² 43 ⁰ C	Decomposition,	Cl' + F',	CO ₂ ,	H ₂ O,	NH ₃ ,	N ₂ + H ₃ , ce STP	NH ₃ , cc STP
2071 ^a	3788	1.47	0.14	ND	48	0.43	0.13	2,14	34.26
2097 ^a	3619	1.41	0.14	ND	52	0,58	0.13	1.83	34.26
2099 ^a	3633	1.63	0.17	ND		0.54	0.16	2.29	42.16
3351 ^b	2641	2.63	0.14	ND	35	0.65	0,14	3.25	73.78
3353 ^b	2641	2,73	0.14	0.02	32	0.77	0.13	3.85	68,52

⁸20 cc of MIL-P-26536B hydrazine.

b40 cc of VL-75 purified hydrazine.

ND = none detected.

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				Specimen			£	Propellant	
	Duration				Weight	Capsule pressure,	Decom -		Halide
Specimen	of test,	Material	Configura- tion	Remarks	change, mg	N/cm- 21 43°C	ромпол. Ж	Remarks	mg
1493	3996	347 CRES	Stug	Light corrosion; thick overall film; light pitting approximately 1 x 10 ⁻⁶ in. diameter.	0.7	10.59	0.57	Slight decomposition: light brown color.	3.70
1499	39%	6061-T6AI	Sing	Light corrosion; thick overall film with cracking in the L/V and V area; light etching and no pitting.	0.8	6.28	0.63	Slight decomposition.	7.83
1527	39%	304L CRES Slug	Sing	Light corrosion; thin film in the L area and thick in the L/V and V areas with cracking in the L/V area; light etching in the L/V and V areas and none in the L area; light pitting in the L area and moderate in the L/V and V areas of about 1 x 10 ⁶ in. diameter.	00	5.54	0.23	Slight decomposition: light yellow color.	0.14
1559	3996	Ti-6A1-4V/ Ti-6A1-4V	Ti-6Al-4V/ Weld, as is Ti-6Al-4V	Light corrosion; thick overall film; light pitting approximately 1×10^6 in. diameter.	6.1	10.00	0.57	Slight decomposition: light yellow color.	0.35
1573	366E	304L CRES 304L CRES	30al CRES Weld, 25 is 30al CRES	Light corrosion; thick overall film with cracking in the L/V area; light etching and no pitting.	8.0	16.19	0.97	Slight decomposition.	0.70
1637	3989	6061-T6 AJ	Slug-stress	No corresion; thin film in the L area and thick in the L/V and a areas with cracking in the L/V area: no pitting.	1.5	92.9	0.23	Slight decomposition.	0.70
1645	3996	Ti-6Al-4V	Slag-stress	Light corrosion in the L/V and V areas and none in the L area; thick overall film; light etching in the L/V and V areas and none in the L area; no pitting.	1.0	10.68	0.55	Slight decomposition. yellow-green colot; dark sediment.	229
1991	3996	304L CRE	304L CRES Slug-strass	No corrosion; thin film in the L and V areas and thick in the L/V area with cracking; no pitting.	0.5	16.62	1.13	Slight decomposition; light brown color.	3.17

Table C-4. Summary of posttest analyses and results — monomethylhydrazine

Table C-4 (contd)

		Halide content, a	0.35
Propellant	Openanic	Democky	light de
4		e. Decomate position,	0.57
		pressure, N/cm ² at 439C	10.16 0.57
		Weight change, mg	-0.2
Specimen		Remarks	Light corrosion; thin overall film with cracking in the L/V area; light etching in the L/V and V areas but none in the L area; light pirting.
		Configura- tion	Slug-stress
		Material	347 CRES Slug-stress
		Duration of test, days	3989
		Specimen of test, number days	1793

*Chloride (habide) introduced as contamination during improper cleaning and preparation procedures. L = liquid exposed region V = vapor exposed region V = liquid/vapor interface

Table C-5. Detailed posttest analyses and results - monomethylhydrazine

	Specim	Specimen weight			₹	alysis of	Analysis of propellant ²					Analysis of gas	of gas		
						Total									
Specimen	Initial,	Change,	Fe.	Ą	Ξ	metal,	Cr+F,	H ₂ 0,	NH3,	Œ	Z Z	EE S	ž		ZH.
number	640	50 0	gur	mg	mg	gm	mg %	18%	22	cc STP	cc STP	cc STP	mole %	mole %	mole ⅓
1493	5.7651	0.0007	0.58	:	:	0.58	3.70	2.07	0.30	0.33	38.69	69.57	35.63	0.30	64.07
1499	1.9680	0.0008	;	1.41	:	1.41	2.82	0.47	0.54	<0.01	11.71	125.23	8.55	<0.01	91.45
1527	5.7548	0.0002	0.37	;	:	0.37	0.14	0.50	0.10	0.08	17.50	23.19	42.92	0.20	56.88
1559	3.8351	-0.0001	:		0.06	90.0	0.35	0.70	0.31	0.13	35.67	71.89	33.12	0.12	66.75
1573	6.7187	-0.0008	1.28	:		1.28	:0.70	0.27	0.53	0.10	62.48	122.91	33.68	0.05	97.99
1637	1.8281	-0.0015		0.48	;	0.48	0.70	0.82	0.07	0.35	23.10	16.23	58.22	0.88	40.90
1645	3.2293	0.0010	:	;	0.37	0.37	2.29	0.16	0.25	0.28	40.05	59.98	39.91	0.28	59.79
1991	5.9349	0.0005	1.95	:	:	1.95	3.17	0.43	0.70	< 0.01	61.05	162.33	27.33	<0.01	72.67
1793	5.8252	-0.0002	1.02	;	;	1.02	0.35	0.38	0.32	0.26	36.22	74.21	32.70	0.23	67.04

²A salysis based on 20 cc of MMH, weighing 17.6 g. -- not measured; data not available.

Table C-6. Summary of posttest analyses and results — nitrogen tetroxide (MON-1)

Specimen							
T comment	of test,		Configura		Weight		NOC
number	- 1	Material	tion	Remarks	change.	O Transfer	content,
2004	4213	347 CRES	Slug	Light corrosion; thin overall film; light etching and no pitting.	0.3		ND ON
0010	3249	347 CRES	Slug	No corrosion; thin overall film; no pitting.	-1.0		ć
. 0012	2443	347 CRES	Slug	No corrosion: thin film in the L/V area and very thin in the L and V areas; no pitting.	0.0		003
9100	3379	6061-T6 Al	Slug	Light corrosion: thick overall film and cracking: light etching and no pitting.	-5.3		0.02
8100	4147	6061-T6 AI	Sing	Light corrosion; thick overall film; light etching and no pitting.	\$40		QN Q
0000	4213	Ti 6Al-4V	Slug	Moderate corrosion; thin overall film: light etching; moderate pitting approximately 1×10^4 in, diameter,	0.6		Q.
0054	3444	Ti 6AL4V	Slug	Moderate corrosion; thin overall film; light etching; moderate pitting approximately 1 x 10 ⁻⁴ in, diameter,	1.2		900
0070	2251	6Al-4V Ti	Slug	Moderate corrosion: thick overall film: light etching. 1 x 10^{-5} in, diameter pitting.	-38.6		0.03
0134	4022	302 CRES	Slug	Light corrosion; thin overall film; light etching; no pitting.	2.		QN QN
0142	3199	302 CRES	Slug	No corrosion; very thin overall film; no pitting	80		6
0146	4147	303 CRES	Slug	Light corrosion; thin overall film: light etching and no pitting.	.		QN ND
0160	4017	304L CRES	Slug	Light corrosion; thin overall film; light etching in the L/V are only; no pitting.	0.7		Š
0162	2251	304L CRES	Slug	Light corrosion; thin overall film; light etching; light pitting of 1 x 10 6 in, diameter,	1.5		<0.03
0178	4147	316 CRES	Slug	Light corrosion; thin film in the L/V and L areas and very thin in the V area; light etching in the L/V and L areas; no pitting.	0.2		Ö.
0180	3255	316 CRES	Slug	No corrosion: thin overall film: no pitting.	0.8		90.00
8170	977	174 PH CRES	Slug	No corrosion; thin overall film: no pitting	1.5	·	<0.03

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	ĭ	ı					origi of P	INAL OOR	PAGE QUAI	: 15 _IT	\$ Y					
Ĕ	NOCI	mg	Q.	<u>R</u>	<0.02	0.03	<0.03		0.0 M		0.03		8 8	S.	<0.03	Q.
Propellant		Remarks								White residue.						
	Weight	mg	1.6	1.2	1.5	9.6-	0.9	1.2	1.9	0.8	0.7	.1.8	-23.4	0.5	0.1	-11.6
Specimen		Remarks	Light corrosion; thin film and light etching in the L/V area; no corrosion, very thin film and no etching in the L and V areas; no pitting.	Light corrosion; thin film and light etching in the L/V area; no corrosion, very thin film and no etching in the L and V areas; no pitting.	Light corrosion, thin film and light etching in the L/V area; no corrosion; very thin film and no etching in the L and V areas; no pitting.	Moderate corrosion; thick overall film; light etching; light pitting of about 1 x 10^5 in. diameter.	Light corrosion; thick overall film; light etching and no pitting.	Light corrosion; thick overall film; light etching and no pitting.	Light corrosion; thick overall film; light etching and no pitting.	No corrosion; very thin overall film; no pitting	No corrosion; very thin overall film; no pitting.	Light corrosion; thick overall film; light etching and no pitting.	Moderate corrosion: thick overall film with cracking in the L/V and V areas: light etching and moderate pitting of about 1 x 10^{-4} in. diameter.	Light corrosion; very thin overall film; light etching in the L/V area only and no pitting.	No corrosion; very thin overall film: no pitting.	Moderate corrosion: thick overall film with cracking: light etching and moderate pitting of approximately 1 x 10^{-5} in, diameter.
		Configura-	Sug	Shug	Slug	Slug	Bimetal STD	Bimetal	Bimetal STD	Bimetal	Bimetal	Bimetal	Slug-stress	Slug-stress	Slug-stress	Weld-stress
		Material	17-4 PH CRES	17-7 PH CRES	17-7 PH CRES	MCKEL	347 CRES	6AI-4V Ti	347 CRES	321 CRES	347 CRES	6061-T6 Ai	Ti 6AL4V	304L CRES	321 CRES	Ti 6A14V
	Duration	of test, days	4017	4017	3199	2196	2196		3199		2237		4009	3968	2237	3968
		Specimen number	0220	0232	0234	0272	0588		0618		0730		96	0980	9560	1016

Table C-6 (contd)

	Pumbion			Specimen		Propellant	=
ecimen umber	Specimen of test, number days	Material	Configura- tion	Remarks	Weight change, mg	Remarks	NOCI content,
1018	3199	Ti6Al-4V/ Ti6Al-4V	Weld-stress	Light corrosson and thin film in the L area; moderate corrosson and thick film in the L/V and V areas; light etching; light pitting in the L/V and V areas only of about $I \times 10^{-5}$ in, diameter.	-13.5		<0.02
1130	3968	304L CRES	Weld	Light corrosion; thin overall film with cracking in the L/V and L areas; light etching and light pitting of about 1 x 10^{46} in, diameter,	03		Q.
1184	2246	6061-T6 AJ	Slug Krytox 240AC	Moderate corrosion: thick overall film: light etching and no pitting.	-1.3	White specks (Krytox residue)	0.03
1404	2246	17-4 PH	Slug Krytox 240AC	Slug No corrosion; thin film in the L/V and V areas and very Krytox 240AC thin in the L area; no pitting.	-0.2	White specks	0.03
1556	4017	N204 ONLY			;		30%

V = vapor exposed region.

L/V = liquid-vapor interface.

--- = not measured, data not available. ND = none detected.

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	Specime	Specimen Weight				An	Analysis of propellant	opellant			
						F		Propet			
Specimen number	Initial, 8	Change, 8	Fe. mg	AI, mg	i,	metal.	NOCI, mg	* t * 8	H ₂ 0.		Parity.
900	5.8655	0.0003	0.46	:	:	0.46	ND ND	33.5	0.08	0970	100.10
0010	5.8166	-0.0010	0.09	÷	:	0.09	0.00	33.5	0.15	0.35	100.9
0012	5.8602	0.0000	0.47	i	:	0.48 (Cu) ^b	0.03	28.88	. :	0.70	80.08
9100	1.8784	-0.0053	;	0.95	:	0.95	0.03	33.5	0.05	0.85	100.16
8100	1.9561	-0.0004	;	1.54	:	1.54	ND	33.5	0.12	0.60	99.75
0000	3.2532	0.0006	;	0.05	< 0.01	0.05	QN.	33.5	0.21	0.51	99.42
0054	3.2258	0.0012	;	0.02	<0.01	0.02	90.0	33.5	0.13	0.58	99.84
0000	3.1796	-0.0386	;	0.14	0.95	1.10 (Cu)	0.03	32.78	:	0.46	99.38
0134	5.8340	0.0014	0.39	;	:	0.39	QN	33.5	0.15	0.67	99.93
0142	5.7152	0.0008	0.10	:	;	0.10	0.02	33.5	0.06	0.50	100.16
0146	5.8333	0.0008	<0.18	;	:	< 0.18	Q.	33.5	0.12	:	99.43
0910	5.8312	0.0007	0.33	:	:	0.33	QN	36.64	0.11	0.52	99.58
0162	5.8578	0.0015	0.31	;	:	0.32 (Cu)	< 0.03	33.08	;	0.33	99.39
8/10	5.8317	0.0002	0.24	÷	:	0.24	QN.	33.5	0.21	0.77	99.65
0810	5.9042	0.0008	0.02	;	:	0.62	0.06	33.5	0.10	0.59	99.64
0218	5.6646	0.0015	0.27	;	;	0.31 (Cu)	<0.03	33.20	:	690	28.97
0220	5.6563	0.0016	0.28	:	;	0.28	Q.	33.5	0.11	0.36	100.10
0232	5.7228	0.0012	0.22	:	:	0.22	QN	35.65	0.16	0.59	100.05
0234	5.7372	0.0015	0.02	,	:	0.02	<0.02	33.5	90'0	0.45	100.20
2720	6.4978	0.00%	11.70	;	:	11.71 (Cu)	0.03	34.46	;	0.63	99.30
0588A	26871	0.0009	0.19	0.04	90.0	0.29	< 0.03	35.20	i	0.63	63.69
0588B	1.4930	0.0012									
0618A	2.6857	0.9019	0.02	:	;	0.02	0.04	33.5	0.05	0.62	100.13
06 18B	2.7263	0.0008									

Table C-7. Detailed posttest analyses and results - nitrogen tetroxide

Table C-7 (contd)

	Specime	Specimen Weight				An	Analysis or proper	penani			
Specimen	Initial,	Change,	Fc.	Al,	T	Total metal. mg	NOCI,	Propellant w.t. ²	H20,	. 8	Parity.
0730A	2.6847	0.0007	0.07	1.47		1.56 (Cu)	0.03	31.71	:	0.67	69.07
0730B	0.9243	0.0018									
9680	3.1650	-0.0234	:	0.06	0.05	0.11	QN	33.78	0.14	0.60	100.00
0650	5.8221	0.0005	0.37	;	:	0.37	Q.	33.5	0.30	0.40	79.65
936	5.8083	0.0001	0.49	:	:	0.50 (Cu)	<0.03	27.52	:	0.64	98.90
9101	3.4109	-0.0116	:	0.03	0.09	0.12	Š	33.5	0.25	0.15	99.56
8101	3.7420	-0.0135	:	0.01	<0.01	0.01	< 0.02	33.5	0.07	0.64	99.88
1130	6.58%	0.0003	0.62	:	;	0.62	Q.	33.5	0.10	0.42	100.00
8	1.9323	-0.0013	÷	2.07	;	2.08 (Cu)	0.03	30.64	;	970	99.04
1404	5,7193	-0.0002	0.95	1	:	0.99 (Cu)	0.03	30.91	:	0.54	99.09
1556	;	÷	0.03	< 0.01	< 0.01	0.03	0.05	35.01	0.12	0.61	99.74

ND = none detected; less than quantity detectable by analytical technique used.

--- = not measured; data not available.

^aPropellant weight:

4 significant figures = accurately weighed during analysis. 3 significant figures = weight estimated.

^hToral metals include those listed in parentheses.

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Surfaces Exposed to Nitrogen Tetroxide

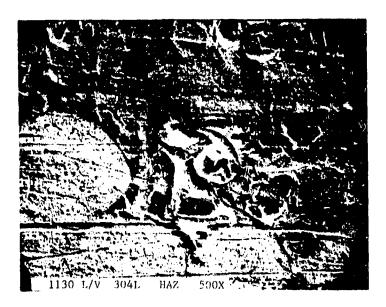


Fig. D-3. Specimen 1130, 304L, heat affected zone, 500X. Thin, irrogular film

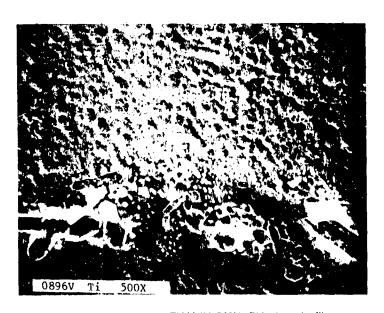


Fig. D-4. Specimen 0896, Ti6Al-4V, 500X. Thin, irregular film, minor cracking and pitting

ORIGINAL PAGE TO OF POOR QUALITY Surfaces Exposed to Monomethylhydrazine

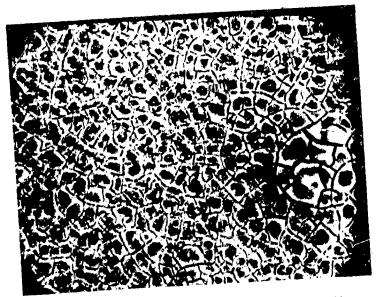


Fig. D-5. Specimen 1499, 6061-T6AI, 500X. Thin film with cracking and flaking

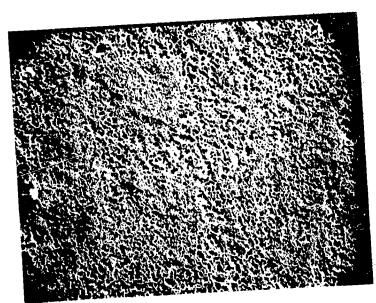


Fig. D-6. Specimen 1527, 304L, 500X. Unbroken surface film

Surfaces Exposed to Hydrazine



Fig. D-7. Specimen 0073, 2014-16AI, 500X. Thin-to-moderate film showing evidence of erosion, not surface pitting

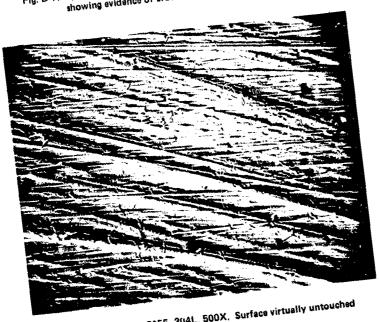


Fig. D-8. Specimen 3155, 304L, 500X. Surface virtually untouched

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Surfaces Exposed to Hydrazine



Fig. D-9. Specimen 1975, 347, 500X. Corrosion at area of highest stress

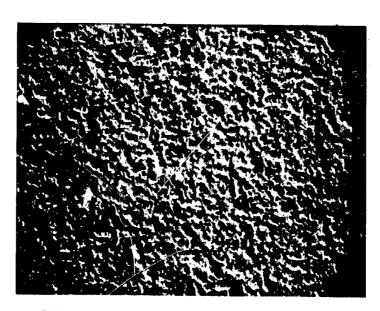


Fig. D-10. Specimen 0191, Ti6Al-4V, 500X. Unbroken surface film